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POTENTIOSTATIC POLARIZATION STUDIES IN FUSED CARBONATES

PART I - THE NOBLE METALS, SILVER, AND NICKEL

by

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POTENTIOSTATIC POLARIZATION STUDIES IN FUSED CARBONATES  
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ABSTRACT

Electrochemical studies in the temperature range 600-700°C are reported for gold, platinum, silver, nickel, and a noble metal alloy, gold-20% palladium in the ternary carbonate eutectic mixture, using the potentiostatic polarization method. Referred to a Ag/Ag<sup>+</sup> reference half cell, the steady-state or corrosion potentials for this series of metals at 600°C are: Au-Pd, -430 mv, Au, -470 mv, Pt, -475 mv, Ag, -680 mv, and Ni, -1166 mv. For nickel at 600°C the anodic potentiostatic polarization curve is characteristic of metal passivation; a primary passive potential is found at -1015 ± 5 mv, but the protection conferred appears far from complete. Further data are reported to contribute to the nature of the anodic and cathodic processes in molten carbonate electrolyses. The phenomenon of carbon deposition at the cathode is reported and examined in light of thermodynamic free energy calculations.

INTRODUCTION

The resistivity to chemical attack of noble metals, silver, and nickel by weight-loss in fused carbonates without applied electrical loads has been recently reported.<sup>1</sup> To gain a further insight on the properties of these metals in contact with such electrolytes, it appeared of interest to undertake potentiostatic polarization studies. While this method is well established in the ambient temperature range<sup>2</sup> for electrochemical studies of metals, use at high temperatures in molten electrolytes appears quite limited<sup>3,4</sup>. The present communication describes the application of this technique for electrochemical studies in the ternary

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$\text{Li}_2\text{CO}_3$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{K}_2\text{CO}_3$  eutectic mixture (mp.  $396^\circ\text{C}$ ), and the results for gold, gold-20% palladium, platinum, silver, and nickel metals in the temperature range  $600\text{--}700^\circ\text{C}$ .

#### EXPERIMENTAL

Potentiostatic Circuit and High Temperature Cell Design: The potentiostat and power supply, commercially available precision transistorised instruments (Model 600 and 620; respectively, Duffers Associates, Inc.), were used with a high input impedance null detector (Model 1230-A, General Radio Company). A five dial Standardizing Potentiometer (Rubicon Company) was used for precise measurement of the electrode potentials; the current was monitored with a Speedomax type G recorder (Leeds and Northrup Company).

The high temperature cell assembly and arrangements are illustrated in Fig. 1a, with details of the test electrode, the auxiliary electrode, and the reference electrode shown in Figs. 1b, c, and d, respectively. The molten carbonate electrolyte was contained in a Au-20% Pd alloy crucible (A); the latter was grounded, contact being made through two Au-Pd wires welded to the crucible and fixed in the transite cover (D, D').

The design of the test electrode, shown in Fig. 1b, consisted of fine metal tubes (0.3 mm dia. x 8 cm lgth; flat closed bottom) joined to a refractory tube (G) used as gas inlet. Electrical contact was made by a wire (E) of the same metal as being studied. This type was used for the Au, Au-20% Pd, Pt and Ag test specimens; for nickel, in addition to the tube, a flat "stick" electrode (4cm x 0.15cm x 30cm) was used. All metals were highest purity commercially available samples. The auxiliary electrode, was designed to take a Pt, Pt-10% Rh thermocouple as shown in Fig. 1c. The gold sheath (L) protecting the refractory thermocouple well was used as the auxiliary electrode in the potentiostatic circuit.

The reference electrode was based on the  $\text{Ag}/\text{Ag}^+$  system, and is of the same design as described by Degobert and Bloch<sup>4</sup> and Danner and Rey<sup>5</sup>. As shown in Fig. 1d, a Ag wire is sealed into the Pythagoras porcelain sheath under 0.5 atm. helium gas; the molten electrolyte (R) in this chamber consists of the  $\text{Li}_2\text{SO}_4, \text{K}_2\text{SO}_4$  eutectic (mp.  $535^\circ\text{C}$ ) containing a small amount of  $\text{Ag}_2\text{SO}_4$ . An outer sheath (P) containing some of the  $\text{Li}_2\text{SO}_4, \text{K}_2\text{SO}_4$  electrolyte (S) protects the inner half-cell assembly from the molten carbonate (which gradually attacks the porcelain). Such assemblies had a useful life of about 30 days in constant use in This Laboratory. Electrical contact with the molten carbonate is through the Pythagoras porcelain which acts as a solid state  $\text{K}^+$  ion conducting membrane. A criterion of the thermodynamically reversible emf. properties of such reference electrodes is the micro-polarization test<sup>6</sup>. The result of such a test of  $600^\circ\text{C}$  is illustrated in Fig. 2. The stability and reproducibility of such electrodes, judged by inter-comparison potentials between a set of two such electrodes, was found highly satisfactory.

The ternary eutectic alkali carbonate mixture ( $\text{Li}_2\text{CO}_3, \text{Na}_2\text{CO}_3$ , and  $\text{K}_2\text{CO}_3$ : 43.5, 31.5, 25.0; mp.  $397^\circ\text{C}$ ) was pre-melted (40 g) in a Au-20% Pd crucible at  $600^\circ\text{C}$  and 1 atm.  $\text{CO}_2$  pressure prior to use. A stream of carefully dried  $\text{CO}_2$  was bubbled through the melt (by means of the test electrode) both to stir the electrolyte and to maintain the  $\text{CO}_2$  pressure well above the thermal  $\text{CO}_2$  decomposition pressure of the ternary melt. The furnace and auxiliary circuit were those described in a preceding study from This Laboratory<sup>7</sup> and it is sufficient to note that the temperature control in the zone was  $\pm 0.5^\circ\text{C}$ .

The steady state "mixed potentials" or corrosion potentials were first gained for the test electrodes relative to the reference electrode by following the cell emf. with the 5-dial potentiometer and the null detector. These are summarized in Table 1. The potentiostatic polarization properties of each specimen were next investigated in the conventional manner<sup>2</sup>. The range of measurements covered

potential from +100 mv and -200 ma to +200 ma at 600°C and 700°C. A series of at least four experiments was made for each metal at both temperatures. Typical results for the anodic studies are illustrated in Fig. 3. The reproducibility of such results is quite satisfactory considering the difficulty of the experimental work at these temperatures.

Control potential electrolyses at high current densities were also undertaken to obtain positive information in the possible nature of the anodic processes for the noble metals. The experimental conditions and results are in Table 2.

Figure 4 shows typical polarization curves for both the cathodic and anodic ranges for the noble metals. After each electrolysis at 600°C, but not at 700°C, a copious black deposit was found in the cathode. This was qualitatively identified by combustion analysis as carbon. Some carbon was also found dispersed in the electrolyte. At 700°C, no carbon (or, at best, trace amounts) was found in the system.

#### DISCUSSION

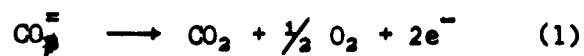
Anodic Electrode Processes: The results show clearly that the alloy, Au-20% Pd, has the most noble value (-430 mv, 600°C, Table 1) of the steady state potentials in the ternary eutectic mixture of lithium, sodium, and potassium carbonates. Arranged in decreasing order of nobility, the series at 600°C for the metals studies is:



where the first three are fairly closely grouped (-430, -470, and -475 mv respectively) with Ag and Ni dropping off to lower values very rapidly (-680 and -1166 mv respectively). Comparison with the recently reported results of Degobert and Bloch<sup>4</sup> shows that the order is essentially the same, but for the noble metals where the order reported is  $\text{Pt} > \text{Pd} > \text{Au}$ . There are insufficient details given in the publication<sup>4</sup> to resolve this difference; it is however apparent that the steady state potentials for the noble metals are also closely grouped as observed in the present work.



The anodic reactions at the noble metal electrodes have been attributed<sup>7</sup> to the carbonate ion oxidation:



and it is of interest to examine the potentiostatic polarization data relative to this and other processes for each metal specifically.

Gold: The corrosion potentials at 600°C and 700°C are virtually identical (-470, -475 mv, respectively, Table 1) and the anodic polarization curves (e.g. Fig. 3) show only a slight shift in this temperature range. The constant potential-high current density electrolyses confirm that less than 0.1% of the current may be attributed to gold oxidation reactions at 600°C and 700°C. If other side reactions are excluded, it is apparent that the current efficiency for the anodic carbonate ion oxidation reaction (1) is greater than 99.9% at gold electrodes in molten carbonates (e.g. 99.97%, 700°C). Use of a gold specimen in similar polarization studies for more than 30 hr. anodic electrolyses at 900°C in molten carbonates gave essentially the same results as with a new and unused specimen (Fig. 3). Microscopic examination of the bright surface of the "aged" electrode showed some appearances of crystallinity, i.e., a pronounced definition of grain boundaries<sup>7</sup>. X-ray diffraction and electron diffraction methods failed to confirm the presence of an oxide film, but the presence of a thin oxide film is not ruled out since the preceding tests were exploratory and of qualitative significance only.

Platinum: The formation of  $\text{Li}_2\text{Pt}_3\text{O}_3$  when Pt is immersed in carbonate melts at 600°C was confirmed in a preceding communication<sup>1,2,7</sup>. In the present experiments it is noted that (i) platinum is significantly less noble at 700°C compared to 600°C (Table 1; corrosion potentials, -475 mv, 600°C; -505 mv, 700°C); (ii) the potentiostatic polarization curves are displaced to higher current densities at 700°C relative to 600°C (Fig. 3); and (iii) the constant potential — high current density electrolyses are in accord with oxide formation (a weight gain) at 600°C; the weight loss at 700°C suggests that the secondary electrode processes may be

platinum metal dissolution or that the metal oxides are unstable at higher temperatures. The preceding thus are additional support for oxide "film" formation on a Pt surface, previously noted<sup>1a,7</sup>, in molten carbonates. The oxide formation appears enhanced at 600°C relative to 700°C, and the change in ohmic resistance accounts for the displacement of the polarization curves (Fig. 3).

It is also significant to note that the primary anodic process in molten carbonate electrolyses with Pt is the carbonate-ion oxidation reaction (current efficiency, greater than 99.96%, 700°C, Table 2).

Gold - 20% Palladium: The potentiostatic results are more similar to Pt than to Au relative to the corrosion potentials (-430 mv, 600°C, -485 mv, 700°C, Table 1) and changes in polarization curves (Fig. 3) with temperature. The evidence for an oxide on the surface of this alloy after immersion in molten carbonates at 600°C has been considered elsewhere<sup>1b</sup>; that the oxide is a palladiate seems not improbable. The present results correlate, as in the case of Pt, with the formation (enhanced at 600°C) of an oxide film on the Au-Pd electrode surface. It is clear that secondary processes to the anodic carbonate ion oxidation at a Au-Pd surface are virtually non-existent in the 600°C-700°C temperature range (Table 2; current efficiency 700°C, greater than 99.99%).

Silver: It is sufficient to note that Ag metal has been reported to be significantly attacked on simple immersion in molten carbonates at 600°C-700°C<sup>1a</sup> or when used anodically<sup>7</sup> in carbonate electrolyses. The present results confirm that Ag metal is quite less noble than Au, Pt, or Au-Pd for such processes (Table 1 and Fig. 2). The corrosion phenomena for Ag in molten carbonates have been discussed elsewhere in detail<sup>1a</sup>; Ag dissolution reactions are enhanced by oxidants (e.g., such as oxygen) that may be present in trace amounts in such melts.

Nickel: The corrosion potential for nickel shows that it is greatly more reactive in molten carbonates than the noble metals (Table 1, Au, -470 mv, Ni, -1166 mv, at 600°C). Inspection of the anodic potentiostatic polarization

curves (600°C, 700°C; Fig. 2) shows that the characteristic pattern of metal passivation at 600°C is found for nickel. A series of experiments at 600°C confirmed that the primary passive potential value occurs at  $-1015 \pm 5$  mv. The protection thus conferred is nevertheless far from complete; inspection of the electrode after such an experiment at 600°C shows that nickel is quite markedly attacked. The phenomenon appears not dissimilar to that noted for Ni metal in molten nitrates<sup>8</sup>.

At 700°C the characteristic passivation loop is not observed but the slope of the potentiostatic polarization curve (Fig. 3) is nevertheless quite close to that at 600°C. After the electrolyses the electrode surface was covered with the same dark film as at 600°C, and the metal was noticeably embrittled. The nature of the corrosion product in such media has been confirmed<sup>1b</sup> as NiO; it is sufficient to note that the anodic attack of nickel in molten carbonate electrolyses appears quite aggressive at 600°C, and is almost extreme at 700°C. This occurs in spite of the tendency to passivation inferred from the potentiostatic polarization results. Thermodynamic interpretation of these data must await a study of the  $\text{Ag}/\text{Ag}^+$  reference electrode relative to the  $\text{O}_2/\text{O}^{2-}$  reference electrode in molten carbonates.

Cathodic Processes: The nature of the primary cathodic electrode process in molten carbonate electrolyses has not been definitely established; it has been generally suggested<sup>9</sup> that the primary process is alkali metal deposition:



with the formation of carbon possibly due to a series of secondary chemical processes involving reaction of the alkali metal with the carbonate or  $\text{CO}_2$ , e.g.,

	$\Delta G^\circ_{900^\circ\text{K}}$ (kcal mol <sup>-1</sup> )	$\Delta G^\circ_{1000^\circ\text{K}}$ (kcal mol <sup>-1</sup> )	
$4\text{M} + \text{M}_2\text{CO}_3 \longrightarrow 3\text{M}_2\text{O} + \text{C}$	2.5	6.5	(3)
$4\text{M} + 3\text{CO}_2 \longrightarrow 2\text{M}_2\text{CO}_3 + \text{C}$	-135	-125	(4)
$2\text{M} + 2\text{M}_2\text{CO}_3 \longrightarrow 3\text{M}_2\text{O} + \frac{3}{2}\text{O}_2 + \text{C}$	212	209	(5)
$\text{C} + \text{CO}_2 \longrightarrow 2 \text{CO}$	2.9	-1.2	(6)

The present data are insufficient for a more detailed analyses of the cathodic processes in molten carbonate electrolyses; further discussion is reserved until additional information has been gained.

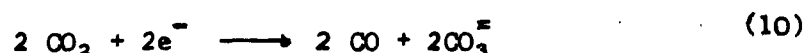
Acknowledgements: Helpful discussions and continued interest of Dr. N. D. Greene, Electrochemical Laboratory, Rensselaer Polytechnic Institute, are gratefully acknowledged. This work was made possible, in large part, by financial support received from the U. S. Navy, Office of Naval Research, Division of Chemistry, Washington, D. C.

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	$\Delta G^\circ_{900^\circ\text{K}}$ (kcal mol <sup>-1</sup> )	$\Delta G^\circ_{1000^\circ\text{K}}$ (kcal mol <sup>-1</sup> )	
$\text{M}_2\text{O} + \text{CO}_2 \longrightarrow \text{M}_2\text{CO}_3$	-45.9	-42.9	(7)
$4\text{C} + \text{M}_2\text{CO}_3 \longrightarrow \text{M}_2\text{C} + 3\text{CO}$	82.2	69.7	(8)
$2\text{M} + \text{M}_2\text{CO}_3 \longrightarrow 2\text{M}_2\text{O} + \text{CO}$	71.5	72.0	(9)

The preceding examples (3-9) illustrate but a few of the reactions that can be thus conceived; the thermodynamic free energy changes,  $\Delta G^\circ_T$  were calculated, using the standard sources of data<sup>10</sup> or estimated values, at two temperatures approximating the experimental conditions, viz 602°C (900°K) and 702°C (1000°K). The formation of carbon by the interaction of ~~the interaction of~~ the alkali metal with CO<sub>2</sub> (4) appears thermodynamically highly favored; the direct reduction of the metal carbonate (3), and the simultaneous oxygen evolution process (5) appear energetically less favorable. The direct cathodic reduction of CO<sub>2</sub> has also recently been proposed<sup>11</sup>:



In combination with the CO<sub>2</sub>-CO-C equilibrium (6) this may lead to carbon formation at proper reaction conditions. It is clear that the mechanism of carbon "deposition" at the cathode in fused carbonate electrolyses is far from understood.

It remains to examine the potentiostatic polarization curves for Au, Pt, and Au-Pd electrodes relative to the experimental observations at 600°C and 700°C respectively (Fig. 4). The anodic branches (all closely identical) of the curves for these metals relate to the carbonate ion oxidation process (1). Inspection shows that the cathodic branches of the polarization curves are also virtually identical for these three metals; this is strong support for the view that the cathodic electrode process is the same at each of these metals (be it alkali metal deposition (2) or direct CO<sub>2</sub> reduction (10)). The presence of carbon at 600°C and its apparent absence at 700°C relates directly to the thermodynamic prediction based on the CO<sub>2</sub>-CO-C equilibrium system (6) in this temperature range. The change in the cathodic polarization values (about 150 mv between 600° and 700°C) may be attributed in part to the CO<sub>2</sub>-CO-C equilibrium process.

### TABLE CAPTIONS

Table 1. Corrosion potentials in the molten ternary eutectic mixture of lithium, sodium, and potassium carbonates.

Table 2. Constant potential electrolyses in the ternary eutectic mixture of lithium, sodium, and potassium carbonates.

### FIGURE CAPTIONS

Figure 1. Electrochemical Cell Assembly and Details of Electrodes for High Temperature Potentiostatic Polarization Studies.

- (a) Electrochemical cell assembly
- (b) test electrode
- (c) auxiliary electrode, and
- (d) reference electrode;
- (A) Au-20% Pd crucible with molten carbonate electrolyte;
- (B) alundum baffles
- (C) stainless steel rods (with refractory beads) and stainless steel baffles (D,D') Au-20% Pd (welded to crucible and grounded,
- (E) gas inlet to test electrode,
- (F) refractory ( $\text{Al}_2\text{O}_3$ ) tube
- (G) Sauereisen cement seal
- (H) welded electrical contact to test electrode
- (I) metal test electrode, with perforations at bottom for gas bubbling,
- (J) Pt-Pt 10% Rh thermocouple
- (K) gold welded electrical contact
- (L) gold sheath (over refractory thermocouple sheath) as auxiliary electrode
- (M) Pt electrical contact wire
- (N) pyrex glass top of reference cell
- (O) Pythagoras porcelain reference half cell body sealed to pyrex
- (P) Pythagoras porcelain sheath to shield the reference half cell from the molten carbonates
- (Q) silver wire
- (R) 0.5 gm of electrolyte of reference half cell (composition: 80%  $\text{Li}_2\text{SO}_4$  - 20%  $\text{K}_2\text{SO}_4$  as solvent, with 0.1 M  $\text{Ag}_2\text{SO}_4$  as solute)
- (S) 1.0 gm. of electrolyte (80%  $\text{Li}_2\text{SO}_4$  - 20%  $\text{K}_2\text{SO}_4$ ).

Figure 2. Micropolarization test for two Ag/Ag<sup>+</sup> reference electrodes (600°C).

Figure 3. Potentiostatic Anodic Polarization Curves at 600°C and 700°C.

(a) Platinum (b) Au-20% Pd alloy (c) Au (d) Ag and (e) Nickel.

(The values of the corrosion potentials at 600°C in the left hand ordinate scale.)

Figure 4. Potentiostatic Polarization Curves at 600°C and 700°C.

The close correspondence of the results for the three metals, Au, Au-20% Pd, and Pt at 600°C and 700°C is shown; the shift in the cathodic branch between the two temperatures corresponds to a displacement of -150 mv.

(700°C)xxx	← Pt;	(700°C)ooo	← Au-20% Pd;	(700°C)aaa	← Au.
(600°C)+++		(600°C)ΔΔΔ		(600°C)nnn	

TABLE 1

Corrosion potentials in the molten ternary eutectic mixture  
of alkali carbonates of Au, Pt, Au-20% Pd, Ag and Ni.

Metal	600 °C	700 °C
	Potential (mv)*	Potential (mv)*
Gold	-470	-475
Platinum	-475	-505
Gold-20% Palladium	-430	-485
Silver	-680	-
Nickel	-1166 mv	-

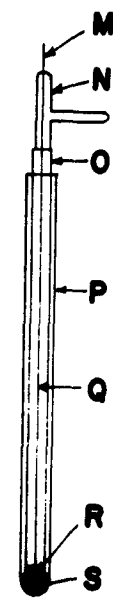
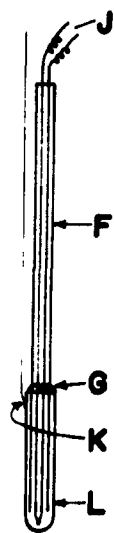
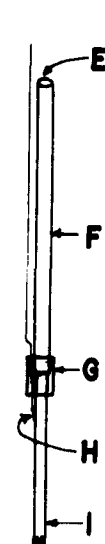
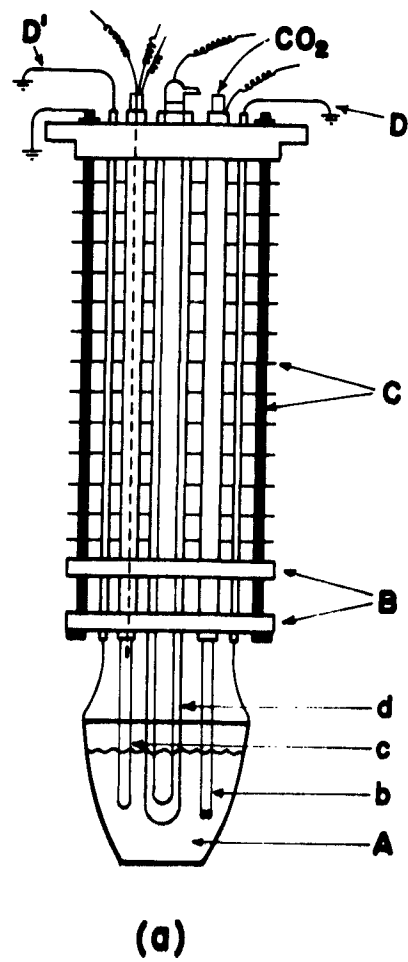
\*Potentials (mv) are relative to the  $\text{Ag}/\text{Ag}^+$  reference electrode as used in this investigation.

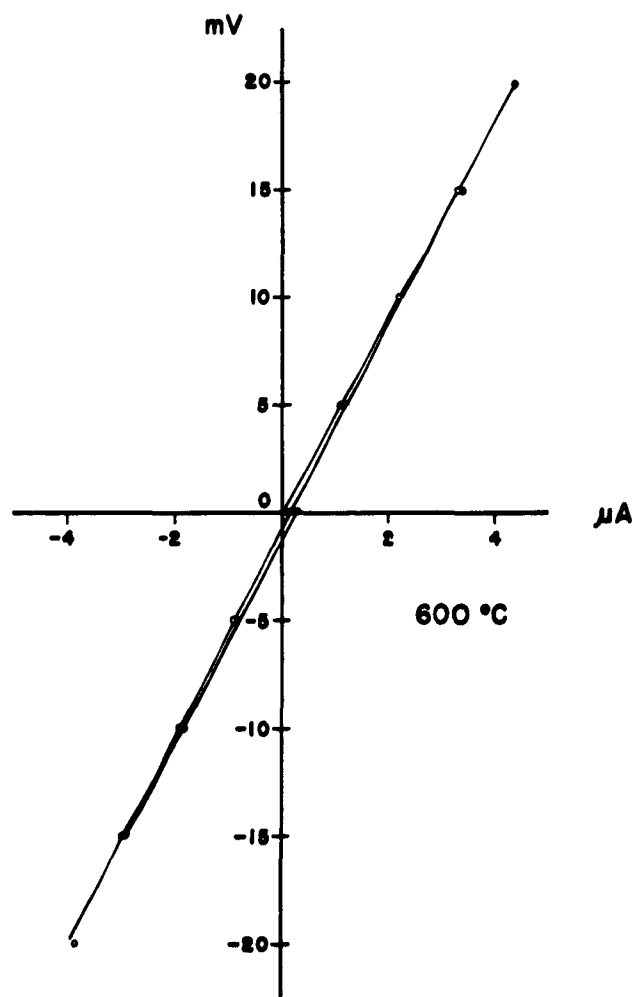


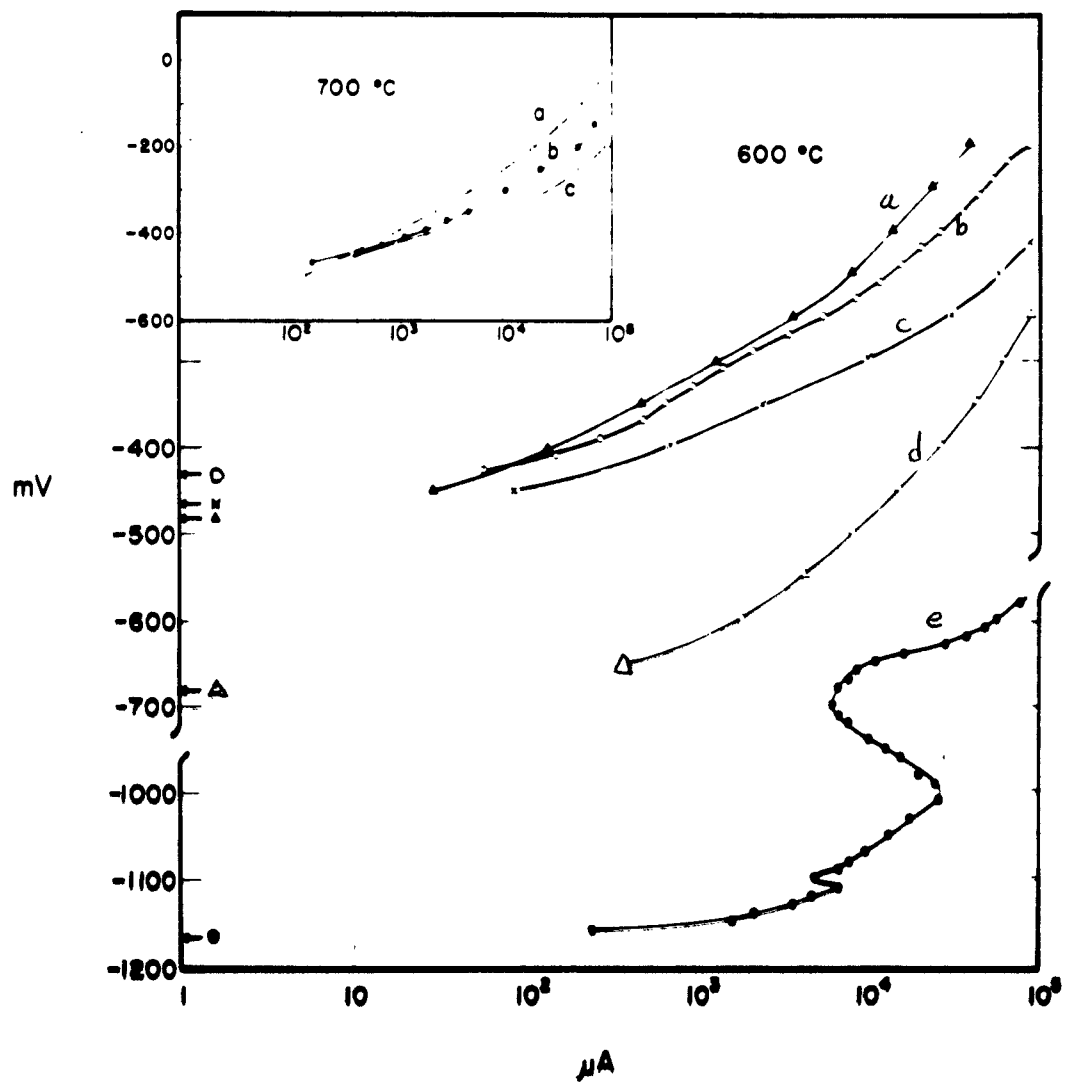
TABLE 2

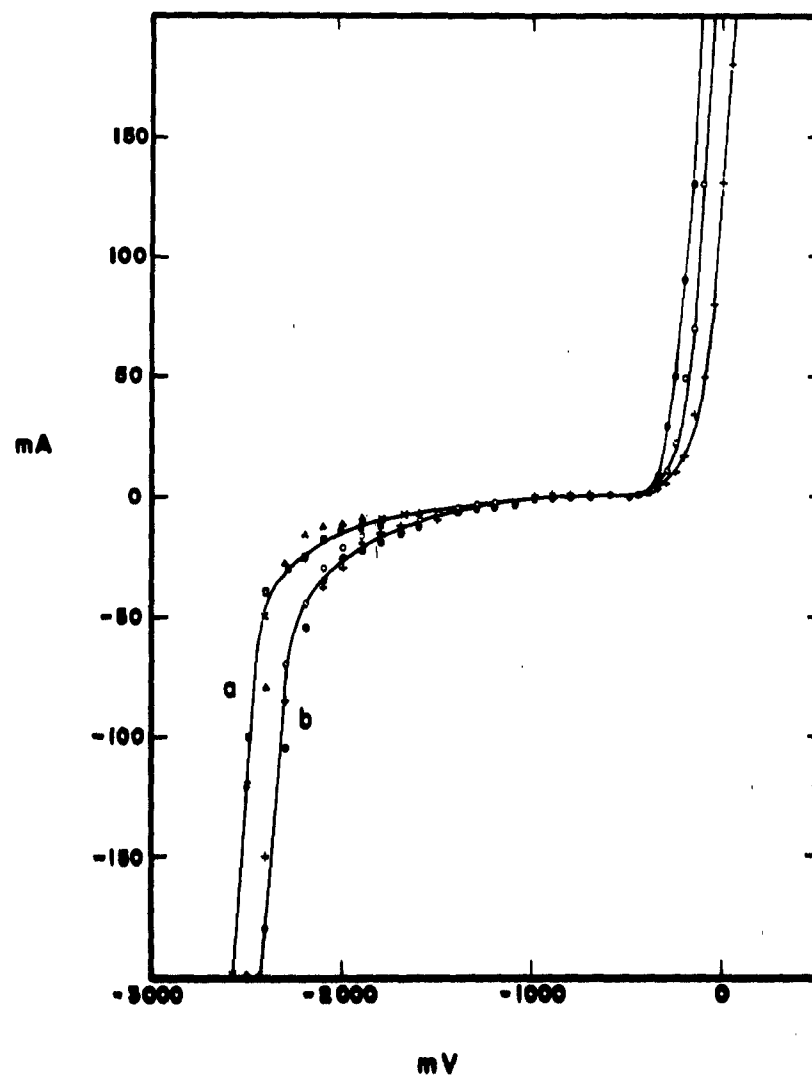
Constant potential electrolysis in the ternary  $\text{Li}_2\text{CO}_3$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{K}_2\text{CO}_3$  eutectic mixture

Anode Material	Temperature (°C)	Time (hrs)	Anode surface (cm <sup>2</sup> )	Potential (mv)	Average c.d. mA/cm <sup>2</sup>	Process Considered	Wgt. change (g)		Metal Dissolution Current efficiency (%)
							obs.	theor.	
Au	600	3	2	-50	83	Au <sup>+</sup>	-0.0007	-3.2	0.02
						Au <sup>3+</sup>	-1.06		0.06
Au	700	3	3	-200	80	Au <sup>+</sup>	-3.65		0.03
						Au <sup>3+</sup>	-1.216		0.10
Pt	600	3	1.26	-100	40		+0.004	- - -	- -
Pt	700	3	1.26	-100	44	Pt <sup>2+</sup>	0.547		0.02
						Pt <sup>4+</sup>	0.273		0.04
Au-Pd (20%)	600	3	1.9	-100	70	Pd <sup>2+</sup>	-3.00		-0.01
						Pd <sup>4+</sup>	-1.50		0.02
Au-Pd (20%)	700	3	1.9	-100	86	Pd <sup>2+</sup>	-3.21		.006
						Pd <sup>4+</sup>	-1.60		0.01









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